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The program consisted of papers on the following topics, aging of polymer glasses, hierarchial structure in biocomposites, electrical properties of self-compositing polymer systems, high performance fibers in compression, damage evolution in polymers and composites prior to fracture, fracture mechanics analysis of matrix cracking in composites, interface of thermoplastics on carbon fibers, composite applications and technologies in advanced aircraft, structure-property relationships in liquid crystalline polymers, carbon fibers from vapor phase hydrocarbons, rheology during gelation, and structure and strength of polymer interfaces.

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PROCEEDINGS

TWELFTH ASILOMAR CONFERENCE ON POLYMERIC MATERIALS

February 12-15, 1989

SPONSORED BY

SUBMITTED BY

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12TH ASILOMAR CONFERENCE
ON POLYMERS

FEBRUARY 12-15, 1989

PACIFIC GROVE, CALIFORNIA

AGING OF POLYMER GLASSES

J. J. Aklonis

It used to be generally believed that once a polymer was taken below the glass transition temperature, all of its properties were essentially fixed. We now know that this is far from being true.

Careful studies of the volume (and the enthalpy) of glasses subjected to simple as well as complex thermal histories have revealed dramatic kinetics. Kovaks, for example, has shown that time dependence, nonlinearity, asymmetry, memory and complicated retardation time effects are characteristic of the behavior of glasses under these conditions. Nevertheless, multiordering parameter models have been developed which essentially quantatively rationalize the observed behavior in a very satisfying manner. Still, one might argue that the changes in dimensions of samples associated with such volume adjustments are of little practical signifigance.

More recently, Struik and others have shown sizeable changes in the mechanical properties of glasses accompaning these small volumetric adjustments. The term physical aging is now commonly used to describe these changes.

We have attempted to generalize the multiordering parameter models which have successfully rationalized volume and enthalpy recovery to treat physical aging. From a qualitative standpoint, our efforts have been successful. However, to date we have been unable to treat both volume relaxation and physical aging within a single consistent framework. We feel that this lack of success is an important indication that the situation is considerably more complicated than it originally seemed.

HIERARCHICAL STRUCTURES

by

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ABSTRACT

The analysis of complex behavior in natural and synthetic polymeric systems in terms of hierarchies is an approach that interrelates our understanding of structure at various scales. Such an approach may prove invaluable in the design of new advanced polymers. Structural hierarchy is more than a convenient vehicle for description and analysis. Hierarchical structure in biological and synthetic composite systems will be described with particular emphasis on organizational levels and the interfacial interactions between such levels.

The lecture is divided into two parts. Firstly, lessons from biology will be discussed with examples of hierarchical structure-mechanical property relations for both reversible and irreversible deformation processes in soft connective tissues. Based on this knowledge and methodology, an oriented multicomposite model showing the overall hierarchical structure will be described from which both the reversible and irreversible mechanical properties can be explained. Then, high performance polymers including liquid crystalline 'molecular' composites and short fiber reinforced thermoplastic composites will be considered. Comparisons will be made between the concepts used in high performance composite structures and the general conclusions derived from naturally occurring composites.

Throughout the lecture the importance of structural organizational levels in controlling mechanical function will be emphasized. Such control is facilitated by the development of unique interfacial processes which is an area of majoi opportunity in the design of polymeric composite systems of the future. Important and difficult questions that remain to be addressed include the physical and chemical factors that give rise to relatively discrete levels of structure and the relations that govern their scaling.

Scattering Studies of Fractions Derived from Linear Low Density Polyethylene and Their Recombinations. .
R. A. BUBECK, and M. A. F. Linne, THE DOW CHEMICAL CO.

A combination of small-angle X-ray (SAXS) and small angle laser light scattering(SALLS) were used to study the morphologies of branch fractions derived from an ethylene-octene copolymer LLDPE. Analysis of the SAXS data featured the use of the Ruland [1] approach to evaluate the interfacial thickness in the transistion zone between the amorphous and crystalline phases. Increasing order in the SALLS spherulite patterns in melt-crystallized fraction samples occurs with decreasing interfacial thickness and increasing branch content. Unbranched linear polyethylene in melt-crystallized form is the most "disordered" from the interfacial viewpoint. A solvent-cast sample always has greater order than its melt-crystallized equivalent. A solvent-blended, melt-crystallized combination of two or three fractions results in a sample with greater disorder than that of the individual components. These and other results indicate that optical properties of PE are influenced not only by average spherulite size but also by their internal order as defined by the interface.
1. W. Ruland, J. Appl. Cryst., 4, 70 (1971)

ELECTRICAL PROPERTIES OF SELF-COMPOSITING POLYMER SYSTEMS
by

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Abstract

Mechanically strong, environmentally stable, and electrically conductive polymeric films or fibers can be fabricated (by dry-jet, wet-spinning) from solutions of poly-p-phenylene benzobisthiazole (PBZT) or poly-p-phenylene terephthalamide (PPTA) with metallophthalocyanine molecular metal precursors (such as nickel phthalocyanine, NiPc). These materials are models for multifunctional polymeric solids resulting from a self-compositing system. A self-compositing system is one in which a desired microstructure is achieved with a multicomponent fluid as a result of the thermodynamic and processing conditions which prevail during solidification. Cyclic voltammetry and controlled potential coulometry indicate that PBZT/NiPc films can be reversibly oxidized to render them highly conductive. X-ray diffraction, electron microscopy, and resonance Raman spectroscopy measurements on example fibers, such as those based on PBZT, indicate a microstructure consisting of small, but well-oriented Ni(Pc)I crystallites imbedded in a uniaxially textured PBZT matrix. It is clear, therefore, that these fibers have their exciting combinations of conductivity and strength/stiffness because their microstructure, which offers both electrical continuity through the oxidized NiPc crystallites and mechanical connectivity through the PBZT fibrils.

HIGH PERFORMANCE FIBERS IN COMPRESSION*

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ABSTRACT

The rapidly increasing usage of advanced fiber composites has led to
structural applications that are compression critical. Despite this
widespread usage, the compressive failure of these materials is poorly
understood due to the possible occurrence of several independent failure
modes. The specific case of lamina compressive failure will be discussed from
a materials perspective. Both fiber- and matrix-dominated failure modes will
be examined. Comparison of the compressive behavior of high performance
polymer, carbon, ceramic and glass fibers and their composites will be made.
Particular attention will be given to conditions that yield fiber failure
versus elastic instability in composites. It will be shown how buckling
instabilities can occur on several levels of structural scale. The inherently
poor compressive strengths of polymer and pitch-based graphite fibers will be
examined in detail. Evidence for compressive failure of these fibers due to
the onset of a microstructural instability will be presented. A potential
solution to this problem of poor compressive strength will be offered with the
introduction of a new class of composite material based on reinforcement with
microfibrillar networks of rigid-rod polymers

Improved Model of Glass Transition Phenomena

by

Suri. N. Ganeriwala and Homer A. Hartung

Conventionally glass transitions in polymers are characterized by a "master curve" and associated "shift factors". This provides qualitative insight into the relationship of rate (or frequency) and temperature effects on the glass transition. The construction of the "master curve" is a tedious process based on the implicit assumption that modulus vs frequency isotherms are congruent with a simple shift.

Recently we obtained data on modulus vs temperature for a synthetic rubber (NBR) using a Fourier Transform Mechanical Analysis (FTMA) apparatus. This is a dynamic mechanical property tester developed in-house. The data showed noticeable spreading of the transition region with increasing frequency. This obviously contradicted the implicit assumption of "master curve" construction and prompted a search for an alternative modeling scheme. At each frequency the modulus vs temperature data could be fitted to sigmoid- shaped modeling function involving five parameters with physical meaning as follows:

        (1)   the mid-point of the transition region
        (2)   the spread of the transition region
        (3)   the height of the transition
        (4)   the intercept below the transition region
        (5)   the slope outside the transition region

Only the first two of these, mid-point and spread, were dependent on frequency and the spread was approximately linear with the mid-point. Mathematically, the height and spread are not independent parameters but both describe different feature of glass transition. The relationship of the mid-point with frequency was treated with the WLF theory.

Treatment of modulus vs temperature data with a five-parameter model is readily accomplished with standard computer systems with non-linear least squares approximations capabilities. This treatment automatically handles both the vertical and horizontal shifts used in manual construction of "master curves". This modeling scheme provides a complete quantitative description of the glass transition and is consistent with the WLF theory.

Damage Evolution in Polymers and Composites Prior to Fracture

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ABSTRACT

The microdeformation and damage mechanisms in polymers and their composites have been studied. Examples include short glass fiber reinforced reaction injection molded (RRIM) polyurethane composites with both a poorly-coupled and well-coupled fiber-matrix interface, unidirectional continuous fiber composites of a thermoplastic polyphenylene sulfide (PPS), and microlayer composites with alternating layers of two immiscible polymers. The mechanical properties in tension or flexure have been related to damage processes, such as matrix cracking, fiber debonding and fiber fracture, as revealed by optical microscopy (OM), scanning electron microscopy (SEM) and acoustic emission (AE). Frequently, techniques are coupled so that damage progression can be monitored during single as well as cyclic loading.

THE STRAIN ENERGY RELEASE RATE OF COMPOSITE MICROCRACKING: A VARIATIONAL APPROACH

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ABSTRACT

A variational mechanics analysis has been used to determine the two-dimensional thermoelastic stress state in cross-ply laminates of type $[0_m/90_n]_s$. The resulting stress analysis has been used to calculate the energy release rate due to formation of a new microcrack in the 90° plies. The analysis accurately includes the effect of residual thermal stresses. When compared with experiments, the new energy release rate expressions are found to predict typical data using a single value for the critical energy release rate for microcracking. This critical energy release rate has a physical interpretation as a microcracking fracture toughness or as an *intralaminar* fracture toughness. Finally, using variational theorems this new microcracking fracture analysis can rigorously be shown to be more accurate than previous attempts at the same problem.

INTERFACE OF THERMOPLASTICS ON CARBON FIBERS

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ABSTRACT

A polycarbonate (PC)/carbon fiber (CF) composite system has been examined with regard to interfacial adsorption and crystallization by altering times and temperatures of annealing. Times up to 180 min and temperatures of 245, 275, and 300°C have been investigated. Transverse tensile, transverse toughness, and scanning electron microscopy results on unidirectional, continuous-fiber composites indicate improved fiber/matrix adhesion at longer times and higher temperatures of annealing. Improvements in transverse toughness and transverse tensile strength of a factor of two is achieved. The data indicate that primarily adsorption rather than secondary interfacial crystallization is the likely mechanism for increased adhesion. Isothermal transverse toughness values have been found to fit well to a Langmuir-type expression. The temperature dependence of adsorption as measured by transverse toughness is described well by an Arrhenius equation. The dependence of transverse toughness on PC molecular weights from $M_w = 26,600$ to $39,800$ was found to be large, with higher molecular weights absorbing more effectively.

COMPOSITE APPLICATIONS AND TECHNOLOGIES IN ADVANCED AIRCRAFT

BY

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Toughness has always ben a consideration when choosing a composite system for an aircraft application; however, until recently, a designer's choices were limited to thermoset matrix systems. Tough thermosets are available but are generally limited to service temperature regimes less than 220°F. Toughness values in thermosets decreases dramatically at high temperature regimes and the fiber modulus of the reinforcing material begins to have a strong effect upon the damage mechanisms. Themoplastics have shown superior toughness to those high temperature thermosets and do not appear to be as strongly affected by fiber modulus. These issues will be discussed in terms of the new ASIP requirements for aircraft.

"STRUCTURE-PROPERTY RELATIONSHIPS OF LIQUID CRYSTALLINE POLYMERS"

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Highly oriented polymeric products have been produced over the past fifteen years by two very different processing routes; from conventional polymers processed to highly oriented extended chain structures, and from "rodlike" polymers which exhibit liquid crystalline behavior. Research has focused on understanding the process-structure-property relationships of these novel materials. The process history of thermotropic LCP's is related to the microstructure frozen into the solid state, i.e., the high mechanical anisotropy of the molecules favors formation of complex skin core and layered structures. The fundamental structures observed in thermotropic liquid crystalline polymers are; 'domains', and microfibrils. Extensive microscopy characterization of the thermotropic copolyesters has resulted in the delineation of a fibrillar, hierarchial structural model which accounts for the structures observed in a broad range of oriented fibers, extrudates and molded articles. Three distinct fibrillar species are observed: microfibrils (50 nm), fibrils (500 nm) and macrofibrils (5 micrometers). Orientation variations are the result of local flow fields on the basic structural units during solidification. Interestingly, the same basic hierarchy is observed for both the lyotropic and the thermotropic LCP's and the microfibrillar structures of all the highly oriented polymers, including polyethylene, appear quite similar.

THE EFFECTS OF POLYVINYL CHLORIDE HIERARCHICAL STRUCTURE ON PROCESSING AND PROPERTIES

by

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ABSTRACT

During suspension polymerization, droplets of polymerizing monomer agglomerate to form 150 μm grains of PVC. PVC precipitates from its monomer to form 1 μm primary particles. The primary particles become the melt flow unit under certain processing conditions. The primary particles have a small microstructure at about 0.01 μm spacing.

PVC's microstructure is mostly amorphous, but it contains crystallites that act as physical crosslinks connected together by tie molecules. This microstructure is responsible for the integrity and persistence of the 1 μm primary particle flow unit in the melt. The microstructure is extended to a larger three dimensional structure by melting during processing followed by recrystallization upon cooling. The strength of this larger three dimensional structure is interpreted from the roughness of extrudate from a zero land length die. This visual method is more sensitive for detecting large three dimensional network structure than measuring capillary entrance pressures, especially for very weak three dimensional structures. Both higher processing temperatures and higher molecular weight PVCs contribute strength to this three dimensional structure. The strength of this large three dimensional structure plays a large role in determining Izod impact and stress rupture.

Plasticized PVC has a structure similar to rigid PVC as judged by the roughness characteristics of its extrudate. In this case, the plasticizer is soluble in the amorphous phase leaving the crystallites intact.

Carbon Fibers from Vapor Phase Hydrocarbons

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ABSTRACT

Production of carbon fibers of macroscopic dimensions from the pyrolysis of hydrocarbons requires two steps. First, carbon filaments of microscopic diameter but appreciable length are produced when nanometer-sized transition metal particles catalytically decompose a hydrocarbon-hydrogen gas mixture, continuously extruding a tubular carbon filament. Such filaments have diameters below 25 nm, yet above 950°C they may lengthen faster than 1 mm/minute. The second stage of the growth process, thickening of the filaments into macroscopic fibers by chemical vapor deposition, accounts for most of the mass of the fibers. Lengthening of these filaments may be understood by applying an adsorption-diffusion isotherm to the surface of the catalytic particle. Observed lengthening rates require only a small carbon supersaturation of the catalytic particle. Filaments have tubular morphology because of the balance between surface energy and stored elastic energy which must be provided as they form.

These vapor-grown carbon fibers exhibit many of the characteristics of pyrolytically deposited carbons; for instance, they have a lamellar microstructure with the graphitic planes exhibiting a high degree of preferred orientation parallel to the fiber axis. Because these fibers are thickened by vapor deposition on catalytically produced thin carbon filaments, they have a cross-sectional structure which resembles the annular rings of a tree. The fibers are so graphitizable that heat treatment to temperatures greater than 2500°C leads to three-dimensional ordering and electronic, magnetic, and thermal transport properties characteristic of single crystal graphite. Due to their microstructure, these fibers offer many potential advantages to the composite materials engineer. These fibers may soon become available as a low priced alternative to conventional carbon fibers.

RHEOLOGY DURING GELATION

by Horst Henning Winter, University of Massachusetts,
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Polymers in the gel state find application as adhesives, super-absorbers, damping materials, i.e. wherever properties in between liquid and solid are required. Processing in the gel state leads to ultimate molecular structures. We, therefore, want to learn more about the properties of gels and want to develop better methods of determining the gel point. Rheology is a powerful tool for these purposes.

At the transition through the gel point, network polymers are in an intermediate state between liquid and solid. Linear viscoelasticity reduces to a simple behavior which is described by the **gel equation** for the stress (J. Rheol., **30**, 367 (1986) and **31**, 683 (1987))

$$\tau(t) = S \int_{-\infty}^{t} (t-t')^{-n} \; \dot{\gamma}(t') \; dt'$$

Molecular parameters determine the front factor, S, and the relaxation exponent, n. The above power law relaxation behavior seems to be a universal property at the gel point. It has been found with a large variety of chemically or physically crosslinking polymers, i.e. with all crosslinking polymers which we studied above the glass transition temperature. The relaxation exponent may theoretically adopt values between 0 and 1, while measured values ranged from 0.15 to 0.8.

The universality of the rheological behavior of polymers at the gel point allows a most accurate determination of the instant of gelation (Polym. Eng. Sci., **27**, 1698 (1987)). The gel point is reached when the complex rheological behavior reduces to power law relaxation in the terminal frequency range (fractal behavior, mechanical selfsimilarity). The novel technique of Fourier Transform Mechanical Spectroscopy (J. non-Newt. Fluid Mech., **27**, 17 (1988)) not only allows the direct determination of the gel point. It also allows, by extrapolation, to predict the distance from the gel point in the pre-gel region. Recent advances of the technique and a set of applications will be given.

STRUCTURE AND STRENGTH OF POLYMER INTERFACES

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The structure of polymer-polymer interfaces is related to the fracture mechanics via deformation mechanisms involving bond rupture and chain disentanglement. The structure of the diffuse interface is described from the static and dynamic properties of chains in melt and the fractal nature of the diffusion front. Fracture of the interface is studied using wedge cleavage and compact tension specimens. Examples are given for welding, fracture and fatigue of several polymer-polymer and polymer-metal interfaces.

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THE RELATION OF THE TENSILE PROPERTIES TO THE MOLECULAR DRAW RATIO OF ULTRADRAWN ULTRA HIGH MOLECULAR WEIGHT POLYETHYLENE

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In this paper, we shall discuss the relation of the tensile properties to the molecular draw ratio of ultradrawn ultra high molecular weight polyethylenes in terms of their macrofibrillar structure, and propose a morphological model which is based on the relation of the tensile properties to the aspect ratio and the shear modulus of the macrofibrils, and which predicts the maximum tensile properties that can be achieved by ultradrawing. According to this model, the highest Young's modulus and tensile strength values that can be obtained are 212 GPa and 13.3 GPa, i.e., significantly close to the theoretically calculated values.

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